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FRACTOGRAPHIC EVIDENCE AND STATISTICAL INFERENCE FOR THE CLEAVAGE FRACTURE MODELLING OF F82Hmod LOW ACTIVATION STEEL

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ABSTRACT

New 7-10% Cr-WVTa steels with reduced generation of long-lived radionuclides following neutron irradiation have been developed during the last years as candidate structural material for components in nuclear fusion reactors. In particular, behaviour in the ductile-to-brittle transition regime is of concern because of the expected shift of the ductile-to-brittle transition temperature following neutron irradiation under in-service conditions.

The paper describes the main results of fractographic investigations performed with notched tensile specimens that were tested at temperatures of −150°C and −75°C and their relation with numerical results for cleavage fracture parameters.

A statistical evaluation of the Weibull stress at fracture was performed following the guidelines of the ESIS P6-98 procedure. Using a novel statistical inference approach, confidence intervals can be obtained for the distribution parameters of the Weibull stress without referring to questionable assumptions about the underlying statistical model. Identification of inhomogeneities in the statistical data is possible and hints on required fractographic investigations for data validation are obtained this way. This is demonstrated using a specific set of results.

INTRODUCTION

Ferritic-martensitic (FM) steels are candidate structural materials for a future fusion reactor. Reduced activation (RA) materials have been developed within the framework of the European Blanket Programme by specific reduction of radiologically unfavourable impurity elements. Additionally, radiologically critical alloying elements (e.g. Mo, Nb) which promote long term activation following neutron irradiation have been substituted by elements with favourable radiological properties (like W, V, Ta) and the Ni content was kept at a low level.

The ductile-to-brittle transition behaviour is of special concern in design considerations because of the observed considerable shift of the ductile-to-brittle transition temperature (DBTT) towards higher temperatures under neutron irradiation.

A fracture mechanics concept based on mechanisms of ductile or brittle failure behaviour is an important tool for the assessment of size and geometry effects, irradiation effects, and effects due to complex mechanical as well as thermal loading conditions. It is particularly necessary for handling the problem of transferability of results from laboratory experiments to component design as well as the problem of the interpretation of results from small specimen testing e.g. under irradiation conditions.

First results of the characterization in the lower shelf regime based on the Local Approach methodology [1,2, 3] were presented in [4]. In the present paper, emphasis will be put on fractographic observations and statistical analysis of the cleavage fracture experiments. After a short description of the experimental procedure and the numerical evaluation of the Weibull stress, results for different notch geometries and temperature levels are given. Typical findings from metallographic investigations can explain most of the results and give additional information with respect to the fracture process. Transferability between results has to be assessed by means of confidence bounds. A novel approach is presented which was already applied within the ESIS TC8 Numerical Round Robin on Micromechanical Models [5]. It uses a Monte Carlo simulation approach and can be used to generate confidence bounds without referring to distribution assumptions. Moreover the method which is known as bootstrap approach [6] permits assessment of the homogeneity of experimental data which is demonstrated in a typical example.

MATERIAL AND EXPERIMENTAL PROCEDURES

The composition of the material is given in Table 1. The material is available in a reference heat treatment condition of 1040°C/38min + 750°C/60min with a DBTT observed in Charpy V tests of about −70 to −50°C [7]. The microstructure is fully martensitic with a grain size of about 70μm and no significant difference in LT, LS and TS orientation.

Table 1: COMPOSITION (WT-%) OF F82HMOD; HEAT NO. 9741

C	Si	Mn	P	S	Cu	Ni	Cr	Mo
0.09	0.11	0.16	0.002	0.002	<0.01	0.02	7.66	<0.01
V	Nb	B	T.N	Sol. Al	Co	Ti	Ta	W
0.16	<0.01	0.0002	0.005	0.001	<0.01	0.001	0.02	2.00

Cylindrical notched bar specimens with a raw diameter of 10mm and a minimum diameter of $d_0 = 5$mm were fabricated from plates of 15mm thickness. The orientation of the specimen axis was perpendicular to the main rolling direction. Notches of 1, 2, and 5mm radius were introduced and surface finish of the notches was achieved by polishing in axial direction.

The tests were performed under displacement control with a crosshead speed of .5 mm/min and continuous optical recording of the minimum notch diameter d. Figure 1 shows results of measured diameter reductions at fracture together with calculated results from large strain deformation plasticity calculations with the ABAQUS Finite Element code [8] that were also basis of the subsequent calculation of the Weibull stress.

NUMERICAL ANALYSIS

The Weibull stress at fracture, σ_W, was calculated from the measured diameter reduction at fracture and the respective stress field obtained by the Finite Element stress analysis. σ_W is defined by

$$\sigma_W^m = \frac{1}{V_0} \int_{V_{pl}} \sigma_1^m \, \mathrm{d}V \qquad (1)$$

where m is the so-called Weibull slope, V_0 is a reference volume introduced for dimensional purposes only and set to 1mm^3, V_{pl} is the volume of the plastic zone, and σ_1 is the first principal stress.

The iterative procedure according to [3] as implemented in the WEISTRABA code [9] was applied to determine the parameters of σ_W, namely the Weibull modulus m and the characteristic value, σ_u. A straightforward calculation leads to the values contained in Table 2, where m- and σ_u-values are given together with their 90% confidence intervals based on the maximum likelihood estimates. Bias correction for m [3] was applied to account for finite sample size.

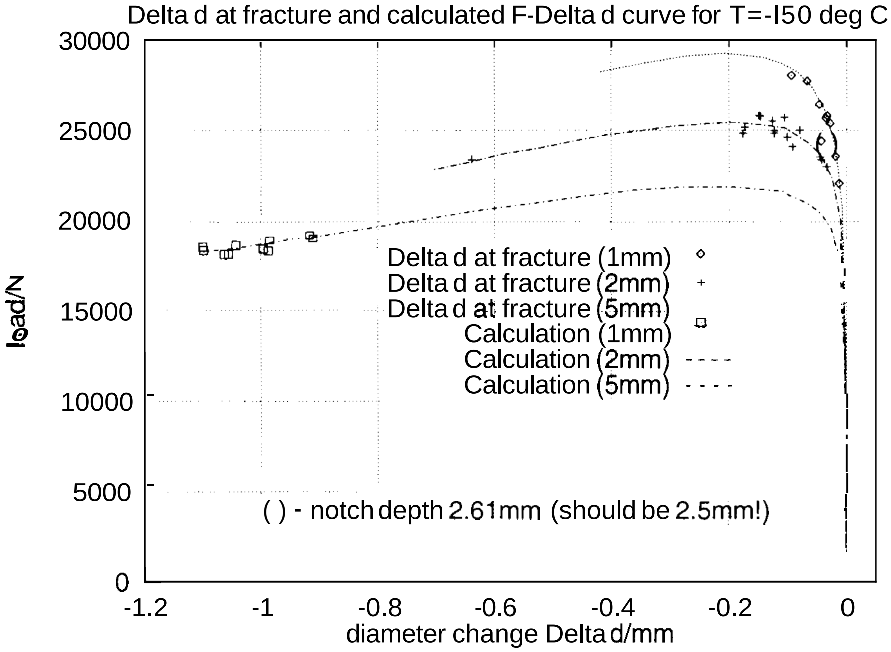

Figure 1: Calculated F – Ad-curves and experimental Ad at fracture.

Two groups of results can be identified. The first group is characterized by moderate values of m (see Weibull plot in Figure 2), whereas in the second group, m attains high values of about 80-100.

All specimens were fractographically examined to reveal possible causes lor the different behaviour within the observed two groups.

For the first group, corresponding to low temperature and small notch radii, fractography showed pure cleavage fracture. Fracture origins could be easily detected as shown in Figure **3.** For the second group, which failed at considerably larger strains, a mixed trans- and intergranular fracture appearance was

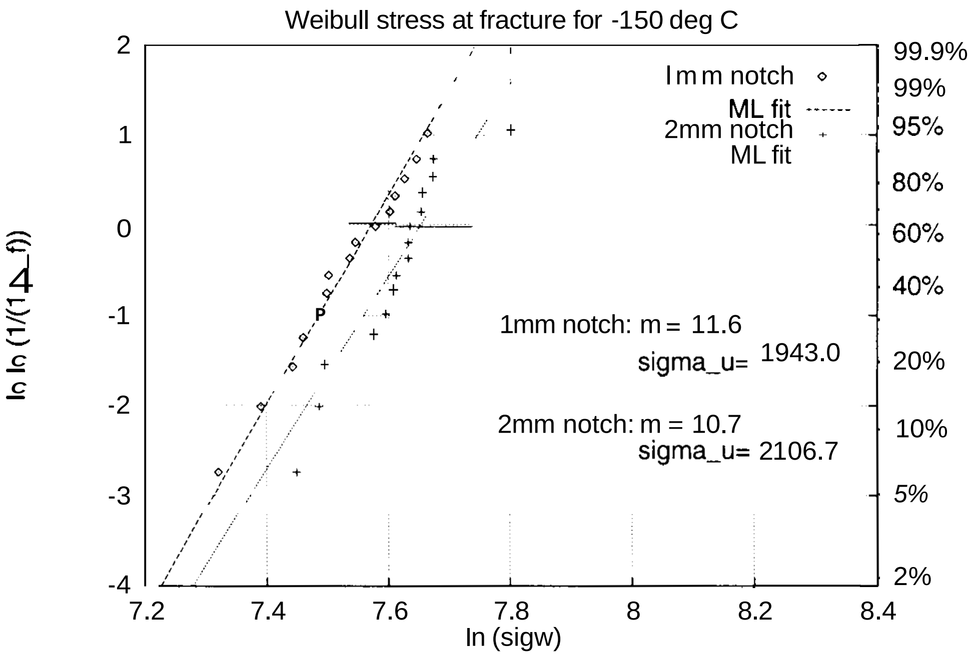

Figure 2: Results of Weibull stress calculation.

Table 2: RESULTS FOR σ_W.

		m	90% ML-CI	σ_u	90% ML-CI
$T = -150°C$	$r = 1mm$	11.6	[8.2, 16.61	1943.0	[1868.6, 2022.01
	$r =$ 2111111	10.7	[7.5, 16.51	2106.7	[2019.3, 2199.71
	$r = 5mm$	78.9	[50.8, 124.51	1913.2	[1899.9, 1927.11
$T = -75°C$	$r = 1mm$	107.5	[69.2, 169.51	1941.1	[1927.9, 1954.71
	$r = 2mm$	78.8	[50.8, 124.31	1764.6	[1752.2, 1777.41
	$r = 5mm$	–	–	–	–

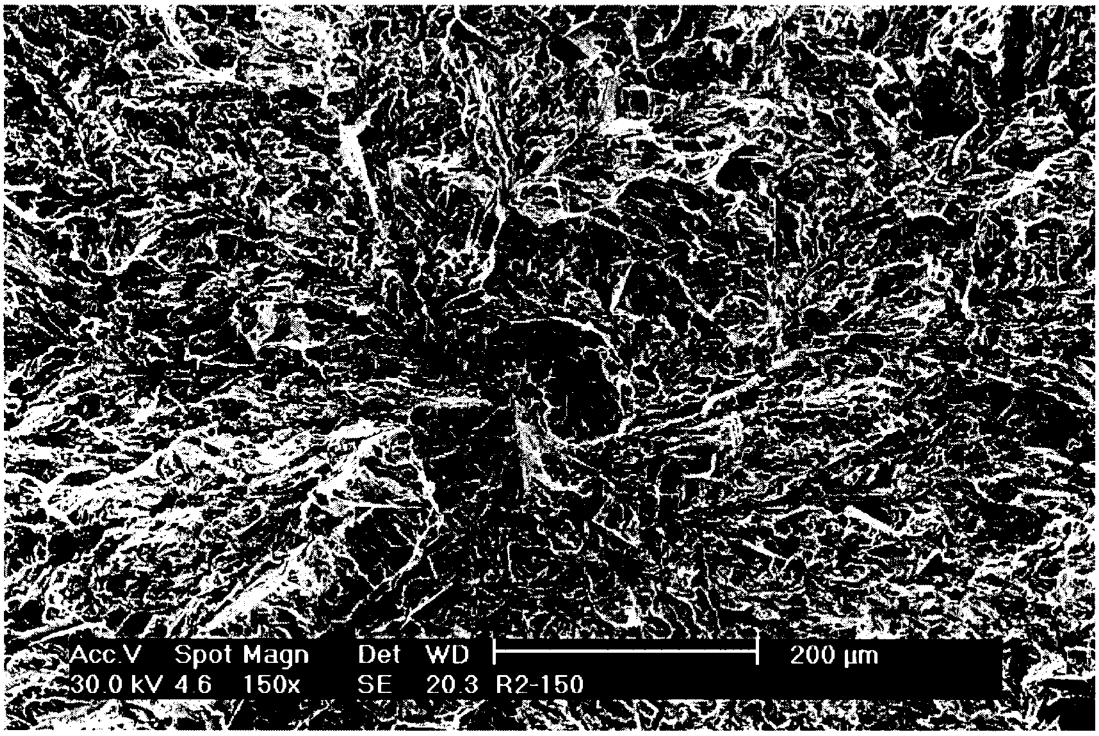

Figure 3: Fracture origin of $r =$ 2111111 notched specimen tested at $T = -150°C$.

observed with indication of ductile damage at the intermediate temperature level of $-75°C$. This is shown in Figure 4, where intercrystalline cracks can be identified in both cases and ductile damage is present for $T = -75°C$.

Additional tests on flat specimens were conducted at room temperature and showed that, with increasing plastic deformation, martensite laths tend to orient themselves in loading direction. This can he seen in Figure 5, where two regions are shown, one located at some distance from the notch, where plastic deformation is negligible, the second region located in the vicinity of the notch. The different orientation of the carbide decorations indicating martensite laths is clearly visible. Change in orientation is connected with shear stresses acting on carbide decorated martensite laths so that finally cracks may be able to initiate. The origin of cracks as shown in Figure **4** is attributed to that kind of shear-controlled crack initiation mechanism.

STATISTICAL ANALYSIS USING THE BOOTSTRAP METHOD

From the preceeding it is clear that statistical analysis alone cannot reveal features that characterize

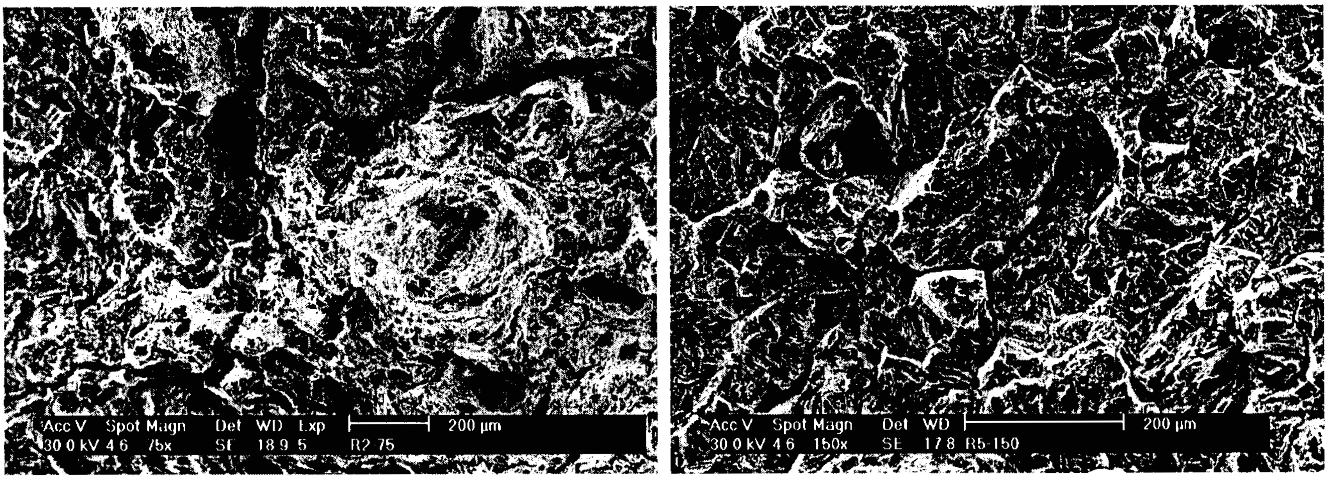

Figure 4: Fracture surface of r = 2mm notched specimen tested at $T = -75°C$ (left) and of r = 5mm notched specimen tested at $T = -150°C$ (right).

the fracture behaviour of a material in a complete manner. However, results can give information on various regions e.g. in temperature and geometry where different mechanisms of damage prevail. It is therefore useful to have methods at hand that allow to trace down inhomogeneities in data sets in order to give hints on planning of subsequent fractographic investigations. A second reason to apply advanced statistical methods as described below was the fact that the Weibull parameters of the Weibull stress are not statistically independent, a fact which is not taken into account by usually applied methods of calculating confidence intervals. One specific data set (r = 2mm notched specimens tested at $T = -150°C$) was selected to demonstrate the use of the so-called bootstrap method. This method was already applied for data analysis within the ESIS TC8 Numerical Round Robin on Micromechanical Models [5].

Basic idea of bootstrapping

Bootstrapping is a statistical inference method based on Monte Carlo simulation [6]. Its basic idea is to use the empirical distribution of the experimentally obtained data as sampling distribution for the generation of a large number of so-called *bootstrap replications*, i.e. repeated calculations of the statistic of interest from the generated bootsprap samples. This is shown in the following scheme:

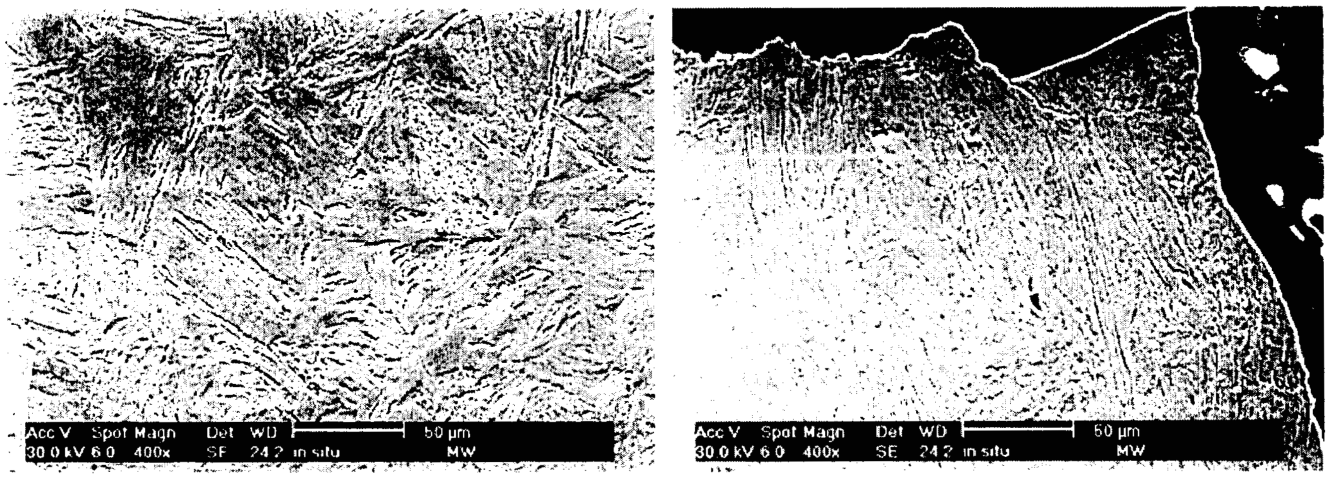

Figure 5: Orientation of martensite laths in undeformed (left) and deformed (right) part of the specimen.

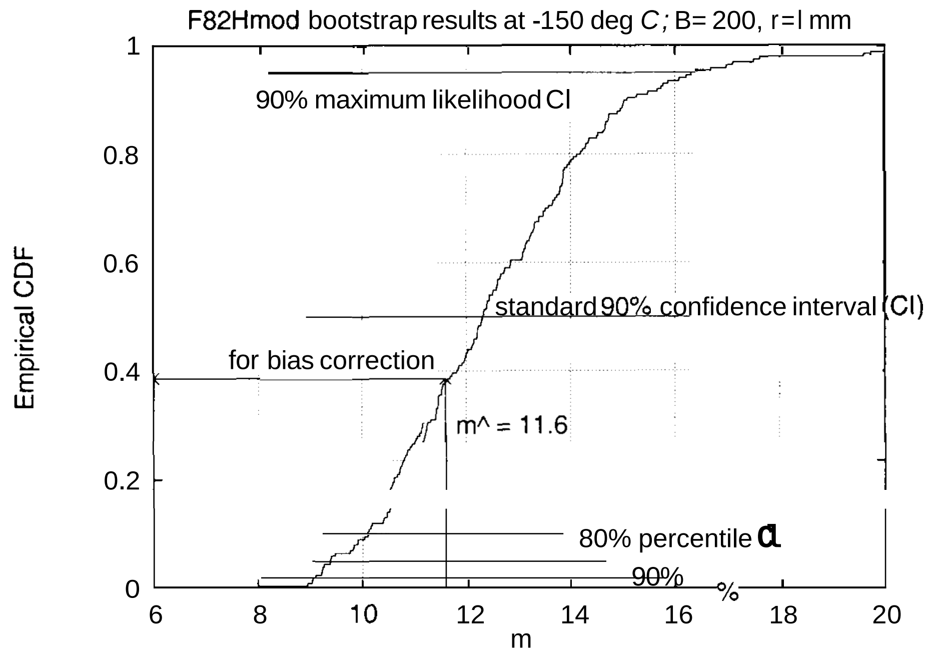

Figure 6: Bootstrap confidence intervals (CI) and 90% maximum likelihood CI.

REAL WORLD			**BOOTSTRAP WORLD**	
unknown probability model	observed data		estimated probability model	bootstrap sample
$P \rightarrow$	$x = (x_1, \ldots, x_n)$ $\downarrow$ $\theta = s(x)$ statistic of interest	$\Longrightarrow$	$\hat{P} \rightarrow$	$x^* = (x_1^*, \ldots, x_n^*)$ $\downarrow$ $\hat{\theta}^* = s(x^*)$ bootstrap replication

A bootstrap estimate of the statistic of interest is obtained as follows:

1. perform random sampling, n times, *with* replacement, from sample x
2. repeat this B times; generate B bootstrap samples $x^{*1}, \ldots, x^{*B}$
3. obtain B bootstrap replications of s, namely $s(x^{*1}), \ldots, s(x^{*B})$
4. obtain a standard error $\widehat{\text{se}}_{\text{boot}}$ for s.

Confidence intervals for s can be obtained by using either the standard error $\widehat{\text{se}}_{\text{boot}}$ for s (leading to *standard bootstrap confidence intervals*) or by using the empirical distribution of the bootstrap replications of s, $s(x^{*1}), \ldots, s(x^{*B})$ (for *percentile confidence intervals*) as shown by the horizontal lines in Figure G for the $r = 1$mm data at $T = -150$°C. A bias correction is possible and uses the value of $s(x) = \theta$ obtained from the experimental sample as indicated in Figure G (here: $\hat{m} = 11.6$). The bias correction is necessary if the empirical CDF value of $\hat{\theta}$ deviates from 0.5 and is the reason for the fact, that the lower bounds of the percentile confidence intervals shown in Figure 6 do not coincide with the empirical cumulative distribution function (CDF) values at the respective probability level but are somewhat shifted to the left.

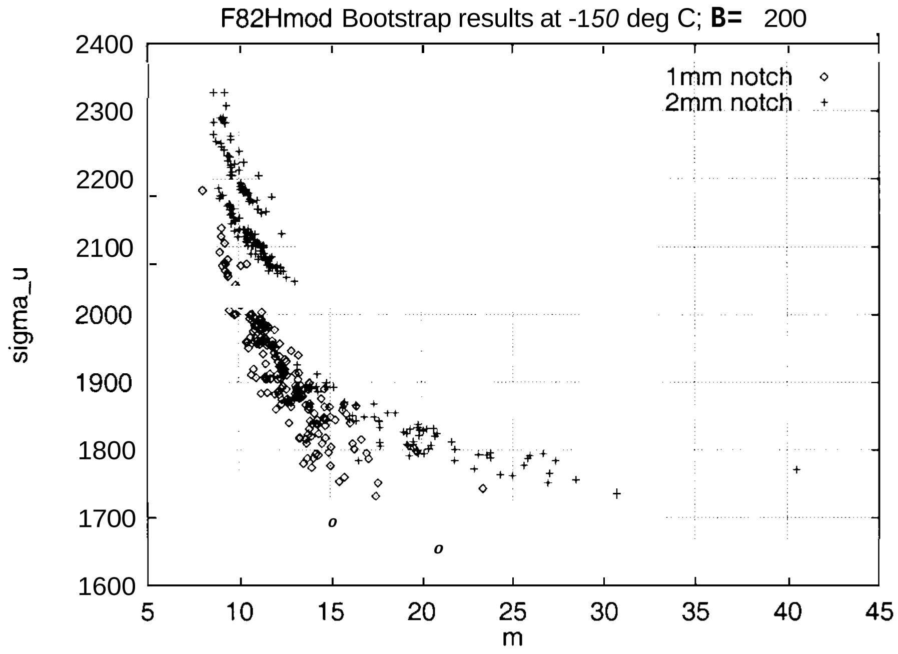

Figure 7: Bootstrap simulation results for (m, σ_u).

Analysis of homogeneity of data sets

Figure 7 shows results of pairs of estimated h'eibull parameters m and σ_u from $B = 200$ bootstrap replications using results of $r = 1$mm as well as $r = 2$mm notched specimens at $T = -150$°C. A strong correlation between m and σ_u is obvious for both cases. For the $r = 2$mm notched specimens, data split into different groups. This can be taken as indication that the sample contains data which is statistically inhomogeneous, i.e. contains one or more outliers. The h'eibull stress calculation results of the two data sets are shown in Figure 2. The Weibull stress at fracture of the last specimen of the 2mm sample is somewhat large, but still well within the confidence bounds. Nevertheless, results of the bootstrap simulations give rise to a closer examination of this specimen by fractography. It turns out that the fracture appearance has similar indications of a mixed trans- and intercrystalline fracture *(see* Figure S) as in the second group. This behaviour could be attributed to an accidental preloading of the specimen combined with insufficient waiting time until the test was conducted so that the temperature

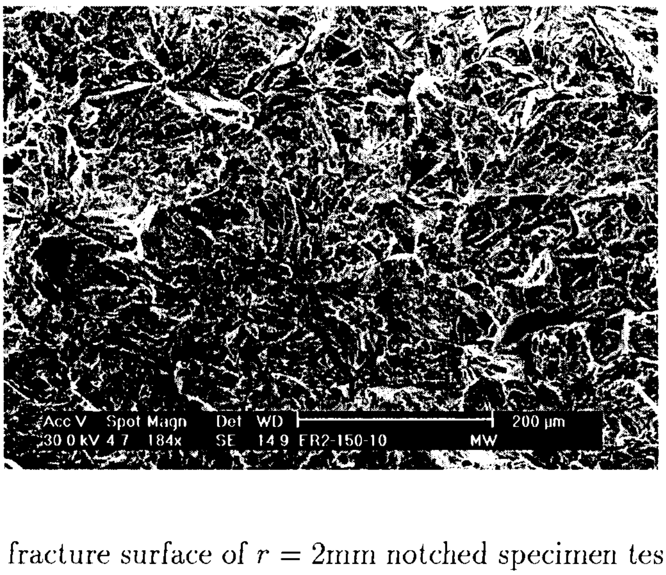

Figure 8: Untypical fracture surface of $r = 2$mm notched specimen tested at $T = -150$°C.

of the specimen was higher than expected.

SUMMARY AND CONCLUSION

Results of numerical and fractographic analysis of F82Hmode cleavage fracture data were presented. Presence of martensite laths in the material led to the formation of microcracks at large local plastic strains leading to high values of the Weibull modulus m both at higher temperatures and for large notch radii where the plastic zone is not confined to the notch root.

The bootstrapping technique for obtaining confidence intervals and additional possibilities for statistical inference was presented. Assessment of data homogeneity by using the bootstrap method *was* shown for a selected set of experimental data.

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